

NANO EXPRESS

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Double-Sided Transparent TiO₂ Nanotube/ITO Electrodes for Efficient CdS/CuInS₂ Quantum Dot-Sensitized Solar Cells

Chong Chen^{1,2*}, Lanyu Ling² and Fumin Li^{1,2*}

Abstract

In this paper, to improve the power conversion efficiencies (PCEs) of quantum dot-sensitized solar cells (QDSSCs) based on CdS-sensitized TiO₂ nanotube (TNT) electrodes, two methods are employed on the basis of our previous work. First, by replacing the traditional single-sided working electrodes, double-sided transparent TNT/ITO (DTTO) electrodes are prepared to increase the loading amount of quantum dots (QDs) on the working electrodes. Second, to increase the light absorption of the CdS-sensitized DTTO electrodes and improve the efficiency of charge separation in CdS-sensitized QDSSCs, copper indium disulfide (CuInS₂) is selected to cosensitize the DTTO electrodes with CdS, which has a complementary property of light absorption with CdS. The PCEs of QDSSCs based on these prepared QD-sensitized DTTO electrodes are measured. Our experimental results show that compared to those based on the CdS/DTTO electrodes without CuInS₂, the PCEs of the QDSSCs based on CdS/CuInS₂-sensitized DTTO electrode are significantly improved, which is mainly attributed to the increased light absorption and reduced charge recombination. Under simulated one-sun illumination, the best PCE of 1.42% is achieved for the QDSSCs based on CdS(10)/CuInS₂/DTTO electrode, which is much higher than that (0.56%) of the QDSSCs based on CdS(10)/DTTO electrode.

Background

Quantum dots-sensitized solar cells (QDSSCs) for converting solar energy directly to electricity have been attracting extensive interest for potential photovoltaic application [1–4]. In QDSSCs, the TiO₂ is widely used as the working electrode due to its non toxicity, high stability, wide availability, and good electronic properties. However, it is known that the TiO₂ mainly absorbs the ultraviolet light due to its large band gap of 3.2 eV. Therefore, various types of quantum dots (QDs) with different optical absorption properties, such as CdS [5–7], CdTe [8–10], CdSe [4, 11–14], PbS [15, 16], PbSe [17], and CuInS₂ [3, 18], have been synthesized to sensitize the TiO₂ in order to extend the light absorption of the TiO₂ into the visible region. To further increase the light absorption of QD-sensitized TiO₂, increasing the loading amount of QDs through the improvement of the TiO₂ photoelectrode structures is

an effective way. Recently, a novel electrode structure, i.e., double-sided transparent TiO₂ nanotube/ITO (DTTO) photoelectrodes were successfully fabricated by our group to enhance light absorption of CdS QD-sensitized TiO₂ photoelectrodes mainly due to the increase of CdS deposition amount [19], in which the TiO₂ nanotube arrays are fabricated on the double-sided transparent ITO substrates. However, for these CdS QD-sensitized DTTO (CdS/DTTO) photoelectrodes, there is still a room for further improvement in light absorption capacity because the CdS/DTTO photoelectrodes mainly absorb visible light with wavelengths less than 550 nm [19]. Hence, for the CdS/DTTO photoelectrodes, there is a prevailing need to find a suitable semiconductor material with a lower band gap than that (2.4 eV) of CdS to harvest more light with wavelengths longer than 550 nm. Copper indium disulfide (CuInS₂) with a narrow band gap of about 1.6 eV is used as the absorption materials in solar cells from its excellent electric and optical properties [3]. Our previous work has shown that the CuInS₂ can be used as a co-sensitizer to extend the spectral response of CdS-sensitized TiO₂

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nanotubes (TNTs) on the Ti substrate into the 500–700 nm wavelength region [18]. Moreover, it has also found that the CuInS_2 can reduce the charge recombination in $\text{CdS}/\text{CuInS}_2$ -sensitized TNTs/Ti electrode. Nevertheless, there is still an issue to be resolved. Due to the opaque Ti substrate, only the QDs deposited on one side of the TNTs/Ti electrode can absorb the sunlight. Obviously, the light-harvesting ability of the opaque TNTs/Ti photoelectrode should be weaker than that of the DTTO photoelectrode.

In this study, we expand our previous work [18, 19]. Considering the advantage of the DTTO photoelectrode in the light-harvesting ability and the complementary effect of CdS and CuInS_2 on the light absorption, the $\text{CdS}/\text{CuInS}_2$ -co-sensitized DTTO photoelectrodes are prepared for the QDSSCs. The detailed synthetic strategy is illustrated in Fig. 1. The surface morphology, optical, and photoelectrochemical properties of as-prepared $\text{CdS}/\text{CuInS}_2$ /DTTO photoelectrodes are systematically studied. The obtained experimental results demonstrate that, compared to the CdS/DTTO photoelectrodes, the light absorption abilities and photoelectrochemical activities of the $\text{CdS}/\text{CuInS}_2$ /DTTO photoelectrodes are increased and the power conversion efficiencies (PCEs) of the QDSSCs based on the $\text{CdS}/\text{CuInS}_2$ /DTTO photoelectrodes are significantly enhanced.

Methods

Materials

Indium tin oxide (ITO, $\leq 15 \Omega/\square$) sheet glass, titanium foil (Ti, Sigma-Aldrich, 0.25-mm thickness, 99.7% purity), ammonium fluoride (NH_4F , Sigma-Aldrich, 98 + %), ethylene glycol (Junsei Chemical Co, 99.0%), cadmium chloride (CdCl_2 , Kanto Chemical Co, 98.0%), indium chloride (InCl_3 , Sigma-Aldrich, 99.999%), sodium sulfide

nonahydrate (Na_2S , Sigma-Aldrich, 98.0%), cupric chloride (CuCl_2 , Junsei Chemical co., Ltd, >97.0 + %), and $\text{Ti}(\text{OCH}_2\text{CH}_2\text{CH}_2\text{CH}_3)_4$ ($\text{Ti}(\text{OBU})_4$, Sigma-Aldrich, 97%). All the materials were used directly without further purification.

Synthesis of Double-Sided Transparent TNT/ITO Films

The TiO_2 nanotube arrays (TNTs) were prepared by electrochemical anodization of the Ti foils. First, the electrolyte consisting of 0.5 wt% NH_4F and 1.5 wt% distilled (DI) water in ethylene glycol (EG) was prepared. Before use, the electrolyte was stirred for 3 h at room temperature. After that, the cleaned Ti foils were anodized at a constant potential of 60 V in prepared electrolyte for 5 h in a two-electrode configuration with a platinum cathode [18]. Then, the formed TNTs were detached from the Ti substrate by intense ultrasonication in DI water. After that, the detached TNTs were adhered onto both sides of ITO glass with a drop of TiO_2 sol. The process for the preparation of TiO_2 sol containing $\text{Ti}(\text{OBU})_4$ and polyethylene glycol has been described in our previous work [19]. Finally, the as-prepared films were annealed at 450 °C for 2 h in air to crystallize the TiO_2 tubes, after which the samples were naturally cooled down to room temperature to obtain the double-sided transparent TNT/ITO films (i.e., DTTO films).

Synthesis of CdS/DTTO and $\text{CdS}/\text{CuInS}_2$ /DTTO Electrodes

CdS and CuInS_2 QDs were deposited on the TNTs by CBD method and SILAR progress, respectively, as described in our previous papers [18, 20]. The CuInS_2 was first deposited on the DTTO films by SILAR progress. The precursors are a 5 mM InCl_3 aqueous solution, a 5 mM CuCl_2 aqueous solution, and a 50 mM Na_2S aqueous solution. The detailed one-cycle synthesis of

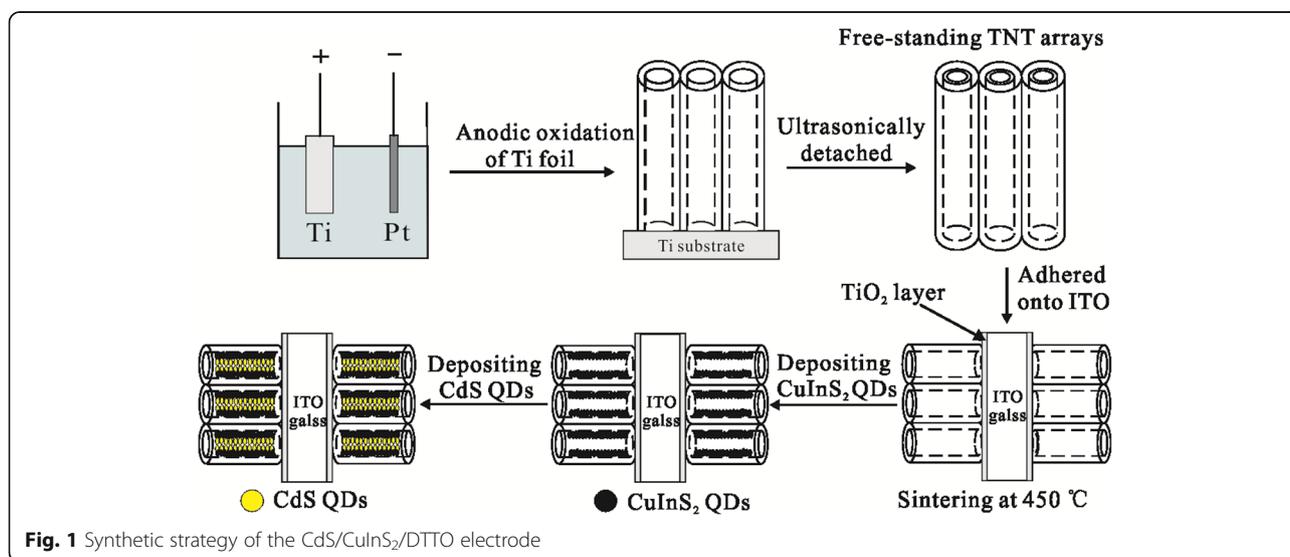


Fig. 1 Synthetic strategy of the $\text{CdS}/\text{CuInS}_2$ /DTTO electrode

CuInS₂ can be obtained from previous publication [18]. In this study, the SILAR cycle was repeated two times.

CdS QDs were deposited on the DTTO and CuInS₂/DTTO electrodes by CBD method. The precursors are a 50 mM Na₂S aqueous solution and a 50 mM CdCl₂ aqueous solution. The electrode was first dipped into 50 mM Na₂S aqueous solution for 1 min, and then rinsed with DI water. After that, the electrode was dipped into 50 mM CdCl₂ aqueous solution for another 1 min, and then rinsed again with DI water. Such a soaking and cleaning process is a typical CBD cycle of CdS deposition. The DTTO and CuInS₂/DTTO electrodes after *n* cycles of CdS deposition are denoted as CdS(*n*)/DTTO and CdS(*n*)/CuInS₂/DTTO, respectively.

Characterization

The SEM images were recorded on a field-emission scanning electron microscopy (FESEM, FEI, Nova230). UV-vis absorption spectra were recorded using a UV-vis spectrophotometer (UV-2550, Shimadzu Corporation, Kyoto, Japan). Transmission electron microscope (TEM) analysis

was done on a Tecnai G2 F30 TEM (FEI Company). Photoelectrochemical reactions of as-prepared samples were carried out in a 250-mL quartz cell, using a two-electrode configuration with the as-prepared samples as working electrode and a Pt counter electrode. A 3-m double-sided adhesive tape sandwiched between the work electrode and the Pt electrode is used to fix the distance between these two electrodes. The photocurrent–voltage characteristics of as-prepared samples with an effective surface area of 0.1 cm^{−2} were recorded using an electrochemical workstation (CHI660E, Shanghai Chenhua Instruments Co., Ltd., Shanghai, China) under simulated AM 1.5G illumination (100 mW cm^{−2}) provided by a solar simulator equipped with a 500 W Xe lamp. The electrolyte was 1.0 M Na₂S aqueous solution.

Results and Discussion

Figure 2a, b shows the top-view SEM image of prepared DTTO film and the cross-section SEM image of the detached TNTs from Ti substrate, respectively. As shown in Fig. 2a, the highly ordered TNTs with an average

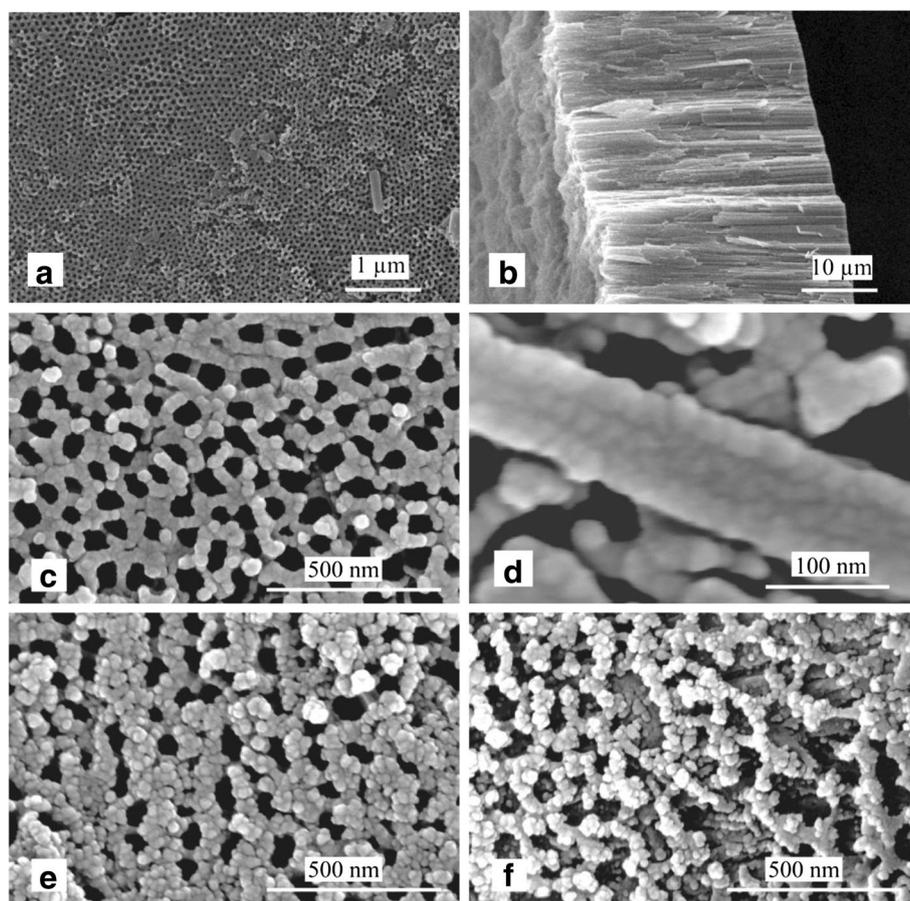


Fig. 2 **a** A top-view SEM image of prepared DTTO film. **b** A cross-sectional view of the detached TNTs from Ti substrate. **c** A top-view image of the CuInS₂/DTTO film. **d** A SEM image of one single CuInS₂/TiO₂ nanotube. Top-view images of as-prepared CdS(10)/CuInS₂/DTTO (**e**) and CdS(15)/CuInS₂/DTTO (**f**) films

inner diameter of ~ 100 nm and a wall thickness of ~ 20 nm are formed. From Fig. 2b, the TNTs with a length up to about $30 \mu\text{m}$ can be observed. Figure 2c displays the top-view image of the $\text{CuInS}_2/\text{DTTO}$ film. It can be observed from Fig. 2c that some CuInS_2 nanoparticles were dispersed on the surface of $\text{CuInS}_2/\text{DTTO}$ film. Moreover, compared to the TiO_2 nanotube in the DTTO film, the inner diameter of $\text{CuInS}_2/\text{TiO}_2$ nanotube decreased slightly due to the deposition of CuInS_2 . Figure 2d displays a SEM image of one single $\text{CuInS}_2/\text{TiO}_2$ nanotube. It can be seen that the CuInS_2 nanoparticles are deposited on the surface of the nanotube and form a CuInS_2 thin film, which is consistent with the reported results [21]. By comparing the inner diameters of TiO_2 nanotube and $\text{CuInS}_2/\text{TiO}_2$ nanotube, it can be obtained that the thickness of the CuInS_2 thin film is about 10 nm. Figure 2e, f shows the top-view images of the $\text{CdS}(10)/\text{CuInS}_2/\text{DTTO}$ and $\text{CdS}(15)/\text{CuInS}_2/\text{DTTO}$ films, respectively. For both films, it can be clearly seen that CdS QDs have been deposited on the TNTs. Furthermore, by comparing Fig. 2e with Fig. 2f, it can be found that more CdS QDs are deposited onto the surface of the $\text{CdS}(15)/\text{CuInS}_2/\text{DTTO}$ film after 15 CBD cycles, indicating that the deposition amount of CdS QDs increases with the cycle number n .

Figure 3a, b shows the low- and high-magnification TEM images of the $\text{CdS}(5)/\text{CuInS}_2/\text{DTTO}$ film, respectively. As shown in Fig. 3a, b, the CdS QDs deposited onto the inner wall of the TiO_2 tube are observed. The average size of the CdS QDs is about 10 nm. Moreover, as shown in the inset of Fig. 3a, the parallel lattice fringes in the wall of $\text{CuInS}_2/\text{TiO}_2$ nanotube are observed. After careful measurement, the interplanar spacing of these lattice fringes is 1.06 nm, corresponding to the (001) plane of tetragonal CuInS_2 (JCPDS 38-0777). The inset of Fig. 3b shows a high-resolution transmission electron microscopy (HRTEM) image of the CdS QDs in the nanotube. The measured lattice spacing for observed fringes is 0.357 nm, which corresponds to the (100) lattice planes of hexagonal CdS (JCPDS 80-0006).

Figure 4a shows the UV-vis spectra of the DTTO, $\text{CuInS}_2/\text{DTTO}$, $\text{CdS}(n)/\text{CuInS}_2/\text{DTTO}$ films ($n = 5, 10,$ and 15). It can be seen that DTTO film absorbs the light with wavelengths less than 400 nm due to the wide bandgap of TiO_2 (3.0 eV). While CuInS_2 are deposited, the absorption spectrum of the $\text{CuInS}_2/\text{DTTO}$ film is extended from 400 to 700 nm, which is consistent with the previous result [18]. Compared to the $\text{CuInS}_2/\text{DTTO}$ film, the absorbance of the spectra of the $\text{CdS}(n)/\text{CuInS}_2/\text{DTTO}$ film significantly increases in the 375–515 nm wavelength region, which can be attributed to the light absorption of CdS. Furthermore, the absorbance of the $\text{CdS}(n)/\text{CuInS}_2/\text{DTTO}$ increases with an increase in CBD cycles, which is mainly due to the

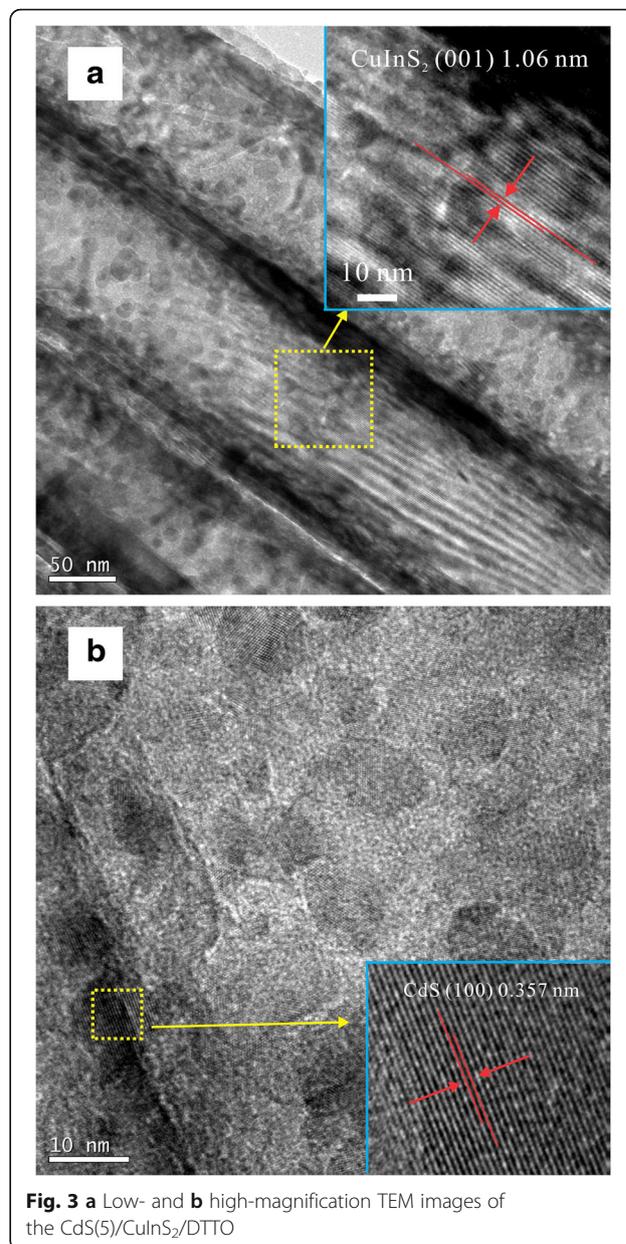
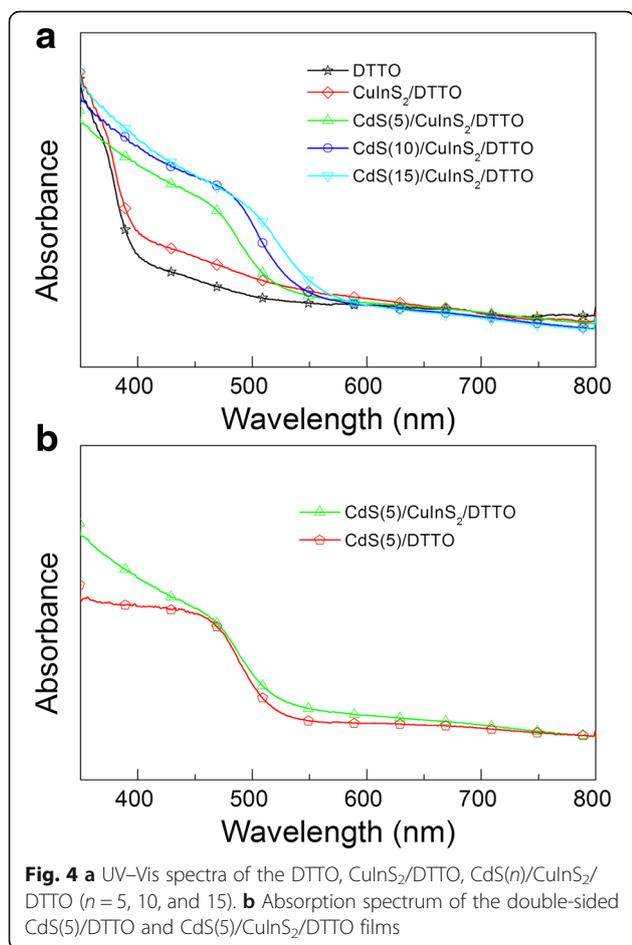


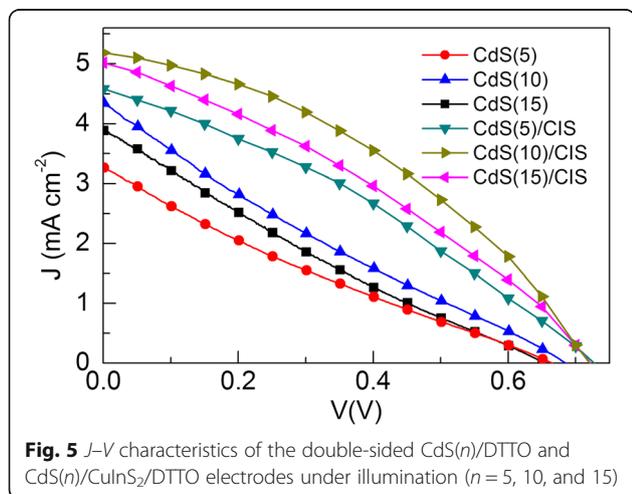
Fig. 3 a Low- and b high-magnification TEM images of the $\text{CdS}(5)/\text{CuInS}_2/\text{DTTO}$

increased deposition amount of CdS. These results are similar to those reported in CdS-sensitized TNTs [20, 22]. To further investigate the effect of the CuInS_2 on the CdS/DTTO film, the absorption spectra of the $\text{CdS}(n)/\text{DTTO}$ and $\text{CdS}(n)/\text{CuInS}_2/\text{DTTO}$ films were measured and compared. As an example, Fig. 4b shows the absorption spectrum of the $\text{CdS}(5)/\text{DTTO}$ and $\text{CdS}(5)/\text{CuInS}_2/\text{DTTO}$ films, which clearly displays that the absorbance of the spectra of the $\text{CdS}(5)/\text{CuInS}_2/\text{DTTO}$ film are enhanced compared with the $\text{CdS}(5)/\text{DTTO}$ film. In particular, the deposited CuInS_2 significantly extended the response of the $\text{CdS}(5)/\text{DTTO}$ into the 500–700 nm wavelength region [22], confirming that the CuInS_2 layer



can effectively improve the light absorption property of the CdS/DTTO films.

Figure 5 shows the *J*-*V* characteristics of the QDSSCs based on prepared CdS(*n*)/DTTO and CdS(*n*)/CuInS₂/DTTO electrodes under illumination (*n* = 5, 10, and 15). Four performance parameters for the measured QDSSCs,



open-circuit voltage (V_{oc}), short-circuit photocurrent (J_{sc}), fill factor (FF), and PCE, have been shown in Table 1.

For the cells based on the CdS(*n*)/DTTO electrodes, the parameters J_{sc} and PCE are increased with the increase of cycle number *n* from 5 to 10 and decreased with the further increase of *n* from 10 to 15. On increasing *n* from 5 to 15, the J_{sc} first increases to 4.35 and then decreases to 3.9 mA cm⁻². This highest J_{sc} (i.e., 4.35 mA cm⁻²) is higher than that of CdS-sensitized TiO₂ nanorod electrode for QDSSCs [23]. Similarly, the PCE first increases from 0.47 to 0.65% and then decreases to 0.56%. The highest PCE of 0.65% is achieved for the cell based on the CdS(10)/DTTO electrode. Moreover, the effects of the cycle number *n* on the values of both V_{oc} and FF are not obvious. These results may be explained as follows: As shown in Fig. 4, the amount of CdS loading increases with the increase of *n* from 5 to 10, which helps to strengthen the light absorption and therefore increase the photocurrent. However, as the cycle number *n* increased further (*n* > 10), the electron-transfer resistance in deposited CdS QDs becomes greater as the loading amount of CdS increases (Fig. 2f) and therefore leads to a more serious charge recombination between the photo-generated electrons in CdS and the redox ions in the electrolyte. Therefore, the J_{sc} , V_{oc} , and FF may decrease with the further deposition of CdS although the light absorption increases.

For the cells based on the CdS(*n*)/CuInS₂/DTTO electrodes with CuInS₂ thin film, it can be seen from Fig. 5 that the effect of the cycle number *n* on V_{oc} is also not obvious. However, the V_{oc} of the cell based on the CdS(*n*)/CuInS₂/DTTO electrode is significantly higher than that of the cell based on the CdS(*n*)/DTTO electrode, indicating that the CuInS₂ can effectively reduce the charge recombination. The parameters J_{sc} , FF, and PCE first increase and then decrease with the increase of *n* from 5 to 15. On increasing *n* from 5 to 10, the J_{sc} increases from 4.6 to 5.18 mA cm⁻², while FF increases from 0.32 to 0.38. As *n* increases further from 10 to 15, the J_{sc} and FF decrease to 5.01 mA cm⁻² and 0.33, respectively. Compared to the cells based on the CdS(*n*)/DTTO electrodes, for a certain cycle *n*, all four

Table 1 Summary of solar cell performances under simulated AM 1.5G solar irradiation

The QDSSCs based on different electrodes	V_{oc} (V)	J_{sc} (mA cm ⁻²)	FF	PCE (%)
CdS(5)/DTTO	0.65	3.27	0.22	0.47
CdS(10)/DTTO	0.66	4.35	0.22	0.65
CdS(15)/DTTO	0.65	3.90	0.21	0.56
CdS(5)/CuInS ₂ /DTTO	0.72	4.60	0.32	1.06
CdS(10)/CuInS ₂ /DTTO	0.71	5.18	0.38	1.42
CdS(15)/CuInS ₂ /DTTO	0.71	5.01	0.33	1.18

parameters V_{oc} , J_{sc} , FF, and PCE of the cells based on the CdS(*n*)/CuInS₂/DTTO electrodes are improved. As shown in Table 1, the highest PCE of 1.42% is obtained for the cell based on the CdS(10)/CuInS₂/DTTO electrode, which is 2.2 times than that (0.65%) of the cell based on the CdS(10)/DTTO electrode. Apparently, the PCEs of the cells based on the CdS(*n*)/CuInS₂/DTTO electrodes have been enhanced largely by the CuInS₂ layer. On one hand, as shown in Fig. 4, optical absorption of the CdS(*n*)/CuInS₂/DTTO electrodes was improved in the wavelengths greater than 500 nm compared to the CdS(*n*)/DTTO electrodes, which would lead to an increased photocurrent. On the other hand, the charge recombination may be reduced through the deposited CuInS₂. For the purpose of facilitating discussion, Fig. 6 shows the energy diagram of the CdS/CuInS₂/DTTO electrode. As shown in Fig. 6, the conduction energy level of CuInS₂ lies between that of CdS and that of TiO₂, which suggests that the photo-generated electrons in CdS can be easily injected into the TiO₂ through the CuInS₂ layer. At the same time, it is difficult for the injected electrons in TiO₂ to recombine with redox ions in the electrolyte because there exists an energy barrier at the interface between the TiO₂ and CuInS₂, which leads to a reduced charge recombination in the CdS(*n*)/CuInS₂/DTTO electrodes and therefore enhances the V_{oc} , J_{sc} , and FF.

Conclusions

In conclusion, the CdS/CuInS₂ quantum dots-sensitized double-sided transparent TiO₂ nanotube electrodes are

fabricated for the QDSSCs. Our experimental results showed that the deposited CuInS₂ enhanced the light absorption of the CdS/DTTO electrodes and reduced the charge recombination in the QDSSCs. These two factors resulted in improved photovoltaic performance of the QDSSCs based on the CdS(*n*)/CuInS₂/DTTO electrodes.

Abbreviations

DTTO: Double-sided transparent TNT/ITO; FF: Fill factor; J_{sc} : Short-circuit photocurrent; PCEs: Power conversion efficiencies; QDs: Quantum dots; QDSSCs: Quantum dots-sensitized solar cells; TNT: TiO₂ nanotube; V_{oc} : Open-circuit voltage

Acknowledgements

We gratefully acknowledge Dr. Guoqiang Li and Dr. Furui Tan for the permission to use the optical measurement system.

Funding

This work was mainly supported by Henan University Distinguished Professor Startup Fund, National Natural Science Foundation of China-Talent Training Fund of Henan (U1404616), Seed Fund of Young Scientific Research Talents of Henan University (CX0000A40540), and Research Fund of Henan University (2013YBZR046).

Authors' Contributions

CC carried out the experiments, participated in the sequence alignment, and drafted the manuscript. FL participated in the device preparation and performed the statistical analysis. LL helped to draft the manuscript. All authors read and approved the final manuscript.

Competing Interests

The authors declare that they have no competing interests.

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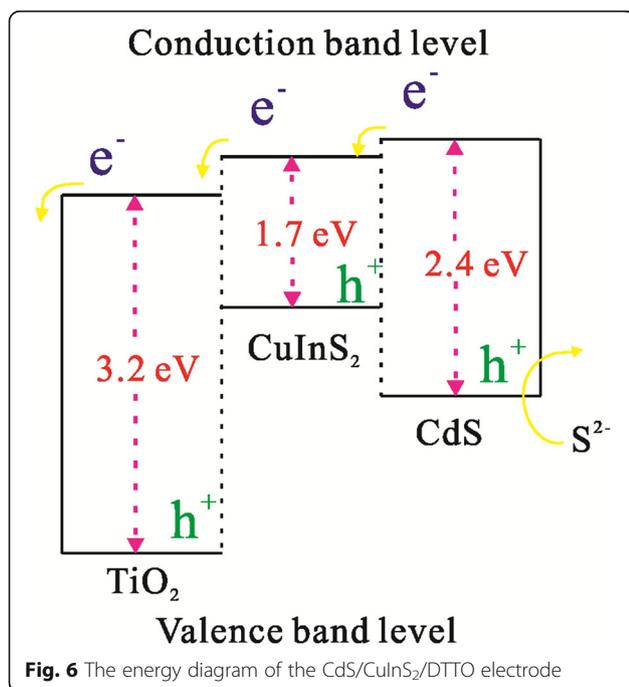
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Received: 14 October 2016 Accepted: 13 December 2016

Published online: 04 January 2017

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